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# HIGH RESOLUTION X-RAY BASED STRAIN MEASUREMENTS FOR HOSTILE ENVIRONMENTS

Final Report on NASA Contract #NAS3-26619
NASA CR-198414 .
October, 1995
Performance Period 2/94 - 6/95

Howard A. Canistraro, Eric H. Jordan and Douglas M. Pease The University of Connecticut Storrs, CT

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#### **ABSTRACT**

A new technique for conducting strain measurements directly through hostile environmental conditions has been developed and tested. The system's unique ability to measure surface strain through conditions such as flame and flowing gases results from the use of focussed, hard x-rays. The x-rays from a molybdenum "Coolidge" type x-ray tube are focussed using a specially designed Bragg reflecting, Johansson ground and bent crystal. Both x-ray tube and crystal are mounted on a linear translation stage, which can scan the focussed image onto prepositioned targets forming a gage length on the test piece. When the focussed image overlaps a target, it fluoresces secondary radiation which is measured. By comparing the known x-ray image position to the fluorescent response of the target, the exact position of the target can be measured relative to the x-ray image position. By using two targets, a gage length is created over which strain is measured. Current tests have been conducted on tensile specimens of Hastelloy-X at temperatures up to 880 degrees C (1600 degrees F), with a repeatability of 0.1 micron (0.004 mil) over a 2.54 cm. (1 in.) gage length. Testing of an advanced silicon nitride composite has also been conducted. The potential for use of this system on components under actual operating conditions is also likely.

#### INTRODUCTION

Strain measurement under hostile environmental conditions is required for the successful development of many newly available materials such as metal matrix (MMC's) and ceramic matrix composites (CMC's). Several techniques currently exist which can conduct these measurements, but they are typically limited to certain environmental conditions such as a vacuum or are limited to around 900 degrees C (1750 degrees F). The goal of this research effort is to produce a non-contacting, high resolution strain measuring device which can function in the presence of intense thermal radiation and "real" atmospheric conditions. The new method exploits the penetrating, non-refractive nature of x-rays such as those emanating from a diffraction x-ray tube.

The fundamental system components are a stable x-ray source, an appropriate focusing crystal which can produce a narrow and intense x-ray line image, a secondary x-ray detector, and fluorescing targets which can be attached to test specimens and withstand the

hostile environment. A typical measurement system is shown in Figure 1. Many difficulties arise when using x-rays due to the fact that they cannot be focused in the traditional manner using mirrors or lenses. However, the use of x-rays is advantageous because they are virtually unaltered by flowing gases, flame or smoke. The investigators have been working under NASA sponsorship (NAG3-1004 and NAS3-26619) and have authored several papers on the topic which describe in greater detail the exact nature and operation of the technique<sup>1,2,3</sup>. The device and principle has also been protected under United States Patent #5,398,273.

The basic concept involves scanning the focused x-ray image onto prepositioned targets which fluoresce secondary radiation. By precisely measuring the focused image position and measuring the secondary radiation from the target, a relative target edge position can be determined by constructing a focused image position-fluoresced radiation overlap curve point-by-point: the focused image is scanned partially onto the fluorescing target and photons are counted for a specific period of time. The image is then moved slightly farther onto the target and again photons are counted. A third point is also taken and a first order curve fit is performed on the points. X-Ray image position is then solved for at some specified secondary radiation level, and is declared the target position. Figure 2 shows the overlap curves resulting from an ideal target scanned at two positions, with the declared overlap value shown. By using two fluorescing targets demarcating a gage length, strain measurement is achieved.

#### **DESCRIPTION OF EQUIPMENT**

A primary goal of the program was the optimization of the system components in order to yield the desired measurement resolution. The two fundamental system variables which are measured are the focused image position, and the secondary radiation from the fluorescing target. Image position measurement is accomplished using a high performance, linear translation stage with an integral mounted laser interferometer capable of measuring table position, and subsequently x-ray image position, with a resolution and repeatability of 0.02 microns (0.0008 mils). Secondary count rate has been optimized by producing the most narrow and intense x-ra, image possible, using a carefully selected xray diffraction tube and a specially designed Johansson ground and bent Si(111) focusing crystal. It should be noted that photon arrival times are random, resulting in an inherent fluctuation in the detected secondary counts (shot noise). This effect leads to a quantum limit of resolution which is illustrated by Figure 3. Therefore, resolution is improved by making the overlap curve as steep as possible. This limit can be reduced further by counting for longer periods of time at each overlap point, at the expense of measurement period. The entire x-ray tube/focusing crystal system is moved as a unit on the linear translation stage as shown in Figure 4. Based on secondary photon/image position overlap curves, resolutions due to quantum limits are on the order of 0.05 microns (0.002 mils) for count periods per point of approximately 0.5 seconds. During the course of development of the technique, several other configurations were used to conduct strain. The performance of these systems ranged from an initial displacement measurement resolution of only 25 microns (1 mil)<sup>2,3</sup>. However, because of improvements made in both the system design and equipment employed, the resolutions have reached this extremely

accurate value. A complete x-ray based strain measuring test facility has been constructed as shown in Figure 5, which is a photograph of the vertical scanning translation table, without the x-ray tube and bent crystal installed. Figure 6 shows the complete scanning system, including the secondary x-ray detector. Software has been developed which automates the scanning process for two fluorescing targets and returns strain information. The program is extremely flexible allowing for many parameters such as count period per point and secondary count levels to be selected.

#### SYSTEM DEVELOPMENT AND TEST RESULTS

Using the original linear translation stage, a repeatability of approximately 0.2 microns (0.008 mils) was temporarily obtained. Unfortunately, the table was not capable of supporting the relatively large overhanging mass of the x-ray tube, bent crystal, and associated apparatus, which can be seen in Figure 6. With the incorporation of the larger, more stable translation stage with the laser interferometer, significant improvements in system resolution and long term repeatability were realized. This system was used to conduct strain measurements at both room and elevated temperatures on both Hastellov X. and a Ceramic Metal Matrix (CMC) composite test specimen. Figure 7 shows the compared results between the x-ray system and an MTS quartz rod extensometer, at 746 degrees C (1375 degrees F) specimen temperature. The limiting factors of this system were the encoder resolution of the linear translation stage of 0.1 micron (0.004 mils), and a somewhat flexible support structure. An additional problem, which became increasingly severe, was a "yaw" error encountered in the translation stage due to bearing wear. It was determined that the table was significantly undersized for the large structure it was intended to support. A new larger table with dimensions of 20.5 cm. (8 inches) wide by 35.5 cm. (14 inches long), increasing the bearing load area and improving overall stiffness. The laser interferometer also aided in improving system resolution. Additionally, the interferometer was mounted away from the center of rotation towards the test specimen, in order to minimize the Abbe' error in image position measurement. The base support structure for the entire system was found to be too flexible to achieve the desired resolution. A 1.5 inch thick (4 cm.) blanchard ground "tool steel" plate was obtained and machined to allow for tilt adjustment and lock-down. Static flexural testing of the new system revealed a deflection at the top of the vertical support bracket of less than 1 micron under a transverse load of over 100 N (23 pounds). The plate on which the x-ray tube and x-ray crystal was also found to be too flexible for the desired accuracy, and it was replaced with a 2.54 cm. (1 in.) thick aluminum tooling plate. These features are shown in Figures 5 and 6. System repeatability, based on a repeated measurement of an unchanged target position; improved dramatically to less than 0.1 micron (0.004 mils) with count periods on the order 0.2 seconds. Figure 8 shows a stress vs. strain curve resulting from a Hastelloy X specimen using the new system at a specimen temperatures of ambient, 738 and 880 degrees C (ambient, 1360 and 1600 degrees F, respectively). The values from the quartz rod extensometer are also shown. The entire process of strain measurement for a single specimen load was reduced to less than 10 seconds through optimization of the computer code.

#### Composite Specimen Testing:

Specimens of Reaction Bonded Silicon Nitride (RBSN), SiC have been obtained from R. Bhatt of NASA Lewis, and have been successfully flame sprayed with Yttria (10% Yttria) Stabilized Zirconia fluorescing targets at Pratt and Whitney Inc. of East Hartford, CT. The flame sprayed targets were machined using a diamond grinding wheel, in order to assure that each edge is parallel with respect to each other. This is necessary for each overlap curve to have similar characteristics for a given focused image alignment. Grips appropriate for these specimens manufactured, using a 6 bolt compression clamp for each end of the specimen.

Room temperature testing of the RBSN specimen showed excellent agreement with both data from an MTS Inc., quartz rod extensometer, as well as expected modulus values provided by Dr. Bhatt. Figure 9 shows a the stress-strain data for the RBSN specimen, in the <100> direction, from both methods. The calculated value of modulus, found from the X-Ray Method data, was 25.4E06 psi, which compares well with the expected range of 24 - 26 million psi. It should be noted that the secondary count rate curve was somewhat less steep due to the shorter fluorescing target length. The original Hastelloy-X specimens were 1 inch wide, with a fluorescing target of similar length. The RBSN specimen was only 0.5 inches wide, and thus, had a shorter fluorescing target length. Repeatability was still on the order of 0.1 microns.

A recrystalized silicon carbide susceptor been produced, similar to that used for inductive heating of ceramics at NASA Lewis by Gary Worthern. The susceptor was made using a cylinder of recrystalized Silicon Carbide, Crystar, which is commercially available from Norton-St. Gobain Inc. The susceptor was then insulated using several layers of ceramic material. The inner layer was a 0.06" Alumina felt, attached using zirconia cement. A 0.06" layer of zirconia was then applied followed by another layer of Alumina. All layers were treated with Alumina rigidizer to increase strength and durability. The susceptor incorporated a "window" to allow the fluorescing targets to be interrogated by the x-ray image. Ports were also fashioned into the "back side" of the susceptor, to allow access by the quartz rod extensometer. Unfortunately, it proved extremely difficult to produce heating of the susceptor, because of the inability to match the impedance of the susceptor to the older Tocco Inc. induction heater. Elevated temperature results were not obtained.

#### Out of Plane Bending Tests:

Sensitivity to out of plane bending of the test specimen can seriously degrade the accuracy and performance of the strain measurement system. It became necessary to test the x-ray method's susceptibility to error resulting from bending of relatively "thin" test specimens. Both the Hastelloy X with a thickness of 0.13 cm. (0.050 in.) and the RBSN Composite specimen with a thickness of 0.25 cm. (0.10 in.) could be considered thin and prone to out of plane bending. In order to ease the assessment of this effect, a large X-Y stage was obtained through a donation from Pratt and Whitney Inc. This stage measured 61 x 61x 23 cm. (24 x 24 x 9 in.), and permitted adjustment of x-ray crystal focal length with respect

to the specimen, as well as lateral alignment of the scanning system. Figure 10 shows a photograph of the X-Y stage incorporated into the x-ray system. The X-Y stage permitted simulated out-of-plane bending testing to be conducted which was achieved by first conducting displacement tests at the proper crystal focal length, and recording the measured gage length between the fluorescing targets. The entire scanning system was then moved incrementally away from the test specimen using the "X" axis of the X-Y stage, and displacement tests repeated. It was found that for movement of the table of up to 127 microns (0.005 in.), no significant change in the measured gage length could be detected. Out-plane-motion in excess of 127 microns (0.005 in.) produced an approximate linear decrease in measured gage length. The sensitivity was found to be roughly 0.5 micron per 25.4 micron (0.02 mil / mil) of out-of-plane motion, beyond an initial scanning system displacement of 125 microns (0.005 in.).

#### **CONCLUSION**

The x-ray system has strain measurement resolution comparable to the best competing systems and unique environmental capabilities. A system resolution of better than 0.1 microns has been achieved, and it's use has been demonstrated in gaseous, hostile environments. Sufficient development has taken place to allow the system to be introduced into the technical community for use as a material property measurement tool in the presence of actual operating environments.

#### References

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- 2. Canistraro H.A., X-Ray Based Displacement and Strain Measurements For Hostile Environments, Ph.D. Dissertation, Univ. of Conn., May 1993.
- 3. Canistraro H.A., Jordan E.H and Pease D.M., X-Ray Based Displacement Measurement for Hostile Environments, Experimental Mechanics, Vol. 32, #4, Dec. 1992.

# AND SECONDARY COUNT TO DATA ACQUISITION SYSTEM X-RAY DETECTOR OVERLAP CURVE OUN T S POSITION T S POSITION T S POSITION T S POSITION SYSTEM

Figure 1. Overall view of typical X-Ray Based Extensometry System.

# **IDEALIZED OVERLAP CURVES OF A TARGET AT TWO POSITIONS**

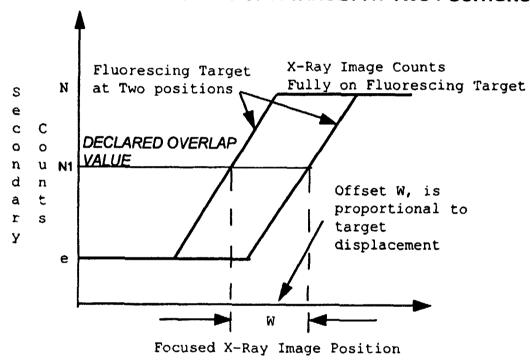


Figure 2: Two overlap curves of an ideal target at two locations, scanned by a uniform, rectangular x-ray image.

#### POSITIONAL NOISE DUE TO POISSON FLUCTUATION Fully on Target S N е c C m = slope of0 0 overlap √N1 N1 = (N - e)/Wn u = dN/dXdn(N) a t $\sqrt{N1}$ = Poisson r s ÿ Fluctuation е dp = positional Off Target noise Noise $= \sqrt{N1/m}$ Floor X-Ray Image Position (X)

Figure 3: Illustration of the quantum limit due to Poisson fluctuation of the secondary count rates, measurement resolution.

# **VERTICAL SCANNING X-RAY EXTENSOMETER**

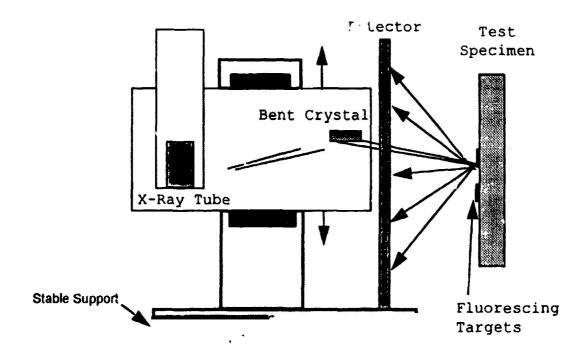


Figure 4: Schematic of the vertical scanning, x-ray extensometer

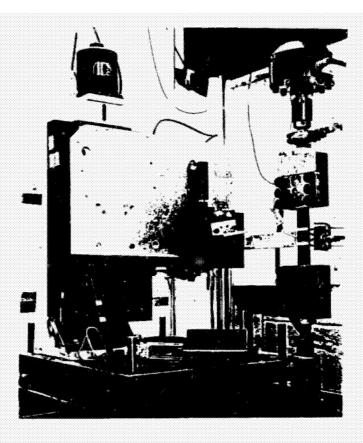


Figure 5: Photo of the scanning system without the x-ray tube, bent crystal and secondary detector installed.

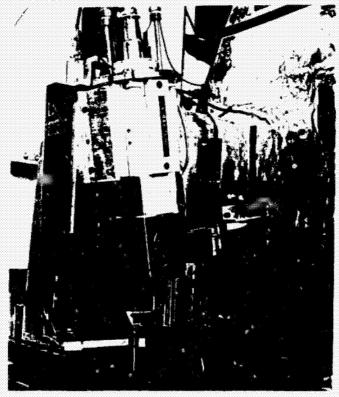


Figure 6. Photo of the complete x-ray extensometry scanning system.

# X-RAY EXTENSOMETER HIGH TEMP. TEST

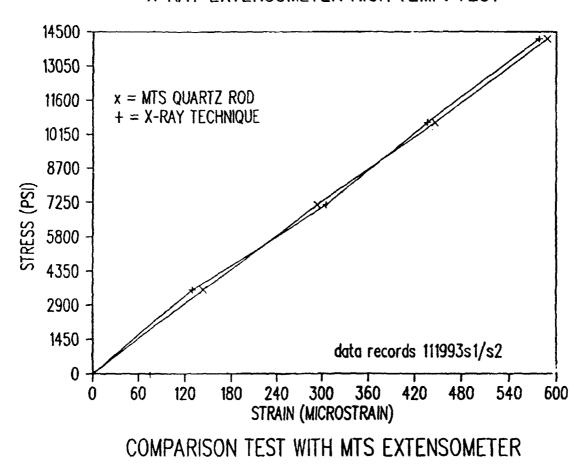


Figure 7: Stress - strain curve from use of the original linear translation stage, of the Hastalloy-X specimen at a temperature of 746 degrees C (1375 degrees F).

# X-RAY BASED EXTENSOMETRY

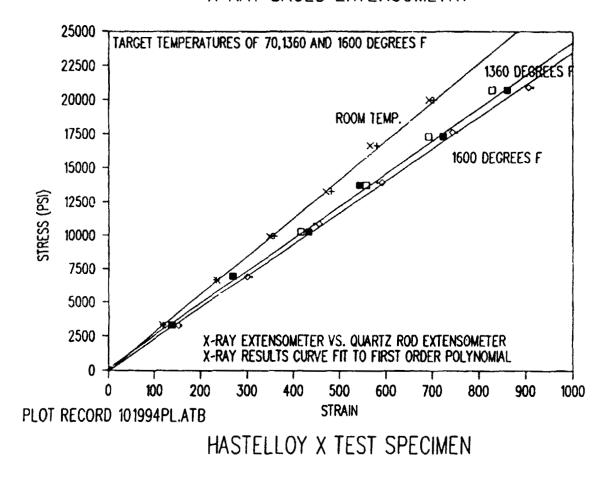
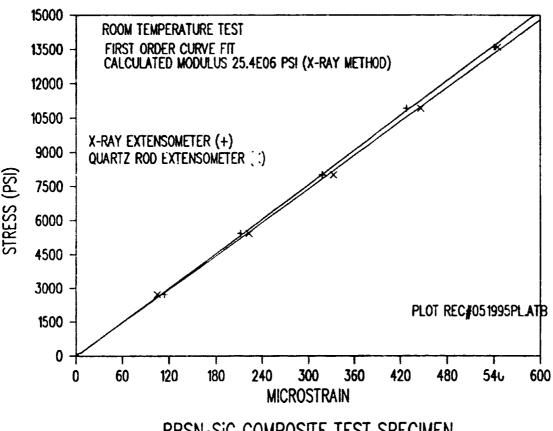


Figure 8: Stress strain curves from use of the new linear translation stage, at specimen temperatures of ambient, 738 and 880 degrees C (ambient, 1360 and 1600 degrees F). The results from the quartz rod extensometer are also shown.

### X-RAY EXTENSOMETRY RBSN SPECIMEN



RBSN-SIC COMPOSITE TEST SPECIMEN

Figure 9: Stress strain curve from a room temperature test of the Reaction Bonded Silicon Nitride, SiC composite test specimer. The results from the quartz rod extensometer are also shown.

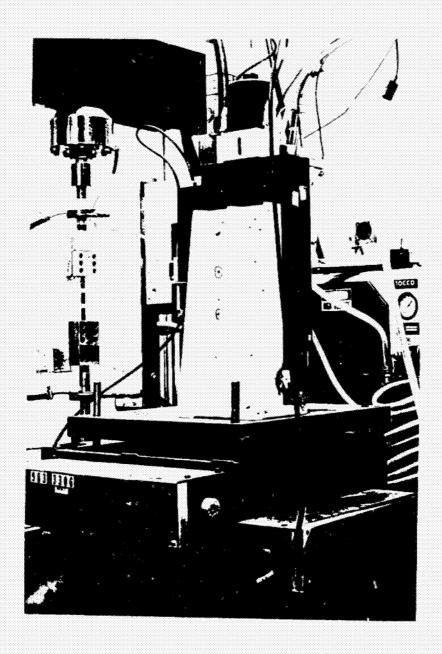


Figure 10: Photograph of the base support X-Y stage, incorporated into the measurement system. This table allows for precise alignment of the x-ray image with the fluorescing target, and adjustment of the Johansson crystal focal length.

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